STANFORD RESEARCH INSTITUTE
MENLO PARK, CALIFORNIA

Monthly Technical Progress Report No. 17 November 15, 1965
October 10 to November 9, 1965

POLYMERS FOR SPACECRAFT HARDWARE--
MATERIALS SPECIFICATIONS AND ENGINEERING INFORMATION

Prepared for: Jet Propulsion Laboratory
California Institute of Technology
Pasadena, California 91103

JPL Contract No. 950745
under NAS7-100

JPL Cognizant Engineers: Robert Harrington
Hugh G. Maxwell

by: R. F. Muraca, N. Fishman,
H. G. Thomas, and J. S. Whittick

SRI Project ASD-5046

Approved: R. F. Muraca, Director
Analyses and Instrumentation

SCOPE

This report covers the work performed during the period October 10 to November 9, 1965 on "Polymers for Spacecraft Hardware--Materials Specifications and Engineering Information," SRI Project ASD-5046 under JPL Contract No. 950745.

The primary objectives of this program are to assist the Jet Propulsion Laboratory in the development and preparation of polymeric material specifications to be used in connection with JPL spacecraft, and to provide a study of the effects of simulated space environment on selected commercial polymeric products. The materials and products to be studied and the extent of work to be performed are specified by the JPL Cognizant Engineers.

The program is being conducted as two interrelated phases, running concurrently: The purpose of Phase I, Polymeric Materials Specifications, is to obtain quantitative values for parameters which may be used to assure the performance of a given batch of material in a spacecraft environment. The purpose of Phase II, Engineering Information, is to establish material limitations and to obtain detailed design information.

PHASE I - POLYMERIC MATERIALS SPECIFICATIONS

WORK PERFORMED

Vacuum-weight-loss determinations by standardized procedures have been made for polyurethane coatings designated as JPL Conformal Coatings-1001 and -1002; formulation of the two coating materials is the same except that JPL-1001 contains a fluorescent dye. The raw materials, packaged as frozen adhesives, were stored at -40°F according to manufacturer's instructions (Ablestik Adhesive Company) since they have a shelf-life of only 24 hours at room temperatures. The adhesives are supplied in plastic syringes containing about 30 grams of material. Preparation of materials for weight-loss determinations was carried out as follows:

> The syringe and contents were warmed to room temperature, and the adhesive was released from the barrel of the syringe onto a glass plate. The plate with the adhesive was placed in an oven maintained at 75°C for the recommended curing period of 4 hours.
>
> The plate and cured coating were then cooled to room temperature, and the coating was released with the aid of a stainless steel spatula.

The resultant coatings were transparent, somewhat tacky, and quite tough. Average thicknesses were as follows: JPL-1001, 0.050"; JPL-1002, 0.065".

The results of the weight-loss determinations are summarized in Tables I and II. The values obtained indicate that these materials may be classified with the better grade of space polymers, i.e., weight losses of 0.5% or less. The values for JPL-1002 appear slightly better than JPL-1001 after 192 hours of exposure in the thermal-vacuum environment; however, the latter achieved an apparent leveling-off within 96 hours, and the former does not show

TABLE I

WEIGHT LOSS DATA FOR
JPL CONFORMAL COATING 1001*
($125^{\circ}C$ and 10^{-6} torr)

Time, Hr	S. Wt., g	Wt. Loss, g	Wt. Loss, %
48	1.6886	0.0060	0.35
	1.7271	0.0067	0.39
96	1.7986	0.0097	0.54
	1.9666	0.0102	0.52
192	2.1083	0.0111	0.53
	2.1922	0.0118	0.54

* Type 2, Lot 2, Batch No. 3-16-66-2

TABLE II

WEIGHT LOSS DATA FOR
JPL CONFORMAL COATING 1002*
($125^{\circ}C$ and 10^{-6} torr)

Time, Hr.	S. Wt., g	Wt. Loss, g	Wt. Loss, %
48	2.1883	0.0061	0.27
	2.2233	0.0066	0.30
96	2.1842	0.0056	0.26
	2.5310	0.0083	0.33
192	2.3954	0.0094	0.39
	2.4697	0.0091	0.37

* Type 2, Lot 1, Batch No. 3-16-66-1

such an indication after 192 hours. On the basis of current data, weight loss limits for JPL-1001 may be suggested as 0.53% $\pm$ 0.05% after a 96-hour exposure period, and for JPL-1002 as 0.38% $\pm$ 0.05% after a 192-hour exposure period.

Weight-loss determinations were also made for circuit board materials supplied by Westinghouse Electric Corporation: Micarta H-8457, an epoxy resin reinforced with fiber glass, 1/8" thick, and Micarta 65M25, a glass-fiber-reinforced epoxy resin clad with copper, 1/16" thick. Samples of the materials were prepared by cutting 1" x $1\frac{1}{2}$" pieces with a band saw.

As shown in Tables III and IV, there is no difference in weight loss between the two materials; thus, a weight-loss limit for both Micartas may be suggested as 0.40 $\pm$ 0.05%.

Maximum weight losses will be corroborated by thermogravimetric analyses in the near future; presently, an auxiliary high-vacuum system is being assembled for operation of the TGA equipment at 10^{-6} torr. The TGA technique will be used also to establish whether high vacuum is really necessary for weight-loss specifications, or whether a simple system operated at 10^{-3} is adequate.*

The literature search has been completed for analytical procedures which will provide meaningful information for specifications purposes for RTV-type silicone adhesives and potting compounds. Recommendations have been made to the JPL Cognizant Engineer for the following chemical and physical tests for the base materials: hydroxyl content, hydrolyzable halide content, filler content, non-volatile content, viscosity, density, and infrared "fingerprint." Additional procedures will include the determination of tin in the

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* Whittick, J. S., "Special Bibliography: Effects of Spacecraft Environments on Polymeric Materials," Stanford Research Institute, Project 4257, January 15, 1965.

TABLE III

WEIGHT LOSS DATA FOR MICARTA H-8457*
($125^{\circ}C$ and 10^{-6} torr)

Time, Hr.	S. Wt., g	Wt. Loss, g	Wt. Loss, %
48	2.7896	0.0094	0.34
	2.7333	0.0086	0.32
96	2.8038	0.0107	0.38
	2.8691	0.0121	0.42
192	2.6558	0.0099	0.37
	2.6714	0.0102	0.38

* Westinghouse Electric Corporation

TABLE IV

WEIGHT LOSS DATA FOR MICARTA 65M25*
($125^{\circ}C$ and 10^{-6} torr)

Time, Hr.	S. Wt., g	Wt. Loss, g	Wt. Loss, %
48	2.9674	0.0107	0.36
	2.9414	0.0103	0.35
96	2.6699	0.0102	0.38
	2.5921	0.0099	0.38
192	3.1972	0.0138	0.43
	3.1281	0.0123	0.39

* Westinghouse Electric Corporation

curing agent (dibutyltin dilaurate), and several tests on cured materials. Work has commenced on the procedure for determination of filler content.

The extended visit of Mr. Maxwell (JPL Cognizant Engineer) during the week of October 25 proved to be invaluable in organizing the contemplated work on the silicones and completing details of the specifications for the epoxy-adhesive series.

FUTURE WORK

Vacuum weight loss determinations via standardized procedures will be made on a continuing basis in order to establish limitations for specifications requirements for various polymeric materials. Corollary studies will be conducted via thermogravimetry in order to verify and supplement the data obtained by standardized procedures.

Determinations of physical and chemical properties of polymers will be continued as required for incorporation into specifications requirements and test procedures.

PHASE II - ENGINEERING INFORMATION

WORK PERFORMED

The fabrication of the 24 VCM "clam-shell" units, which will provide engineering information on volatile condensable materials, has been completed and the units are ready for assembly on a multiply-branched support to be contained within a bell-jar. However, this effort has been halted, momentarily, in order to expedite the fabrication of a multiple unit which will provide within 24 hours values of the maximum VCM for 12 materials--a rather dramatic illustration of the interrelationship of the two phases of this program.

The new design will accommodate micro-size samples and should provide both VCM and weight-loss data. Since the sample weights will be of the order of milligrams, this VCM value will reflect the maximum amount of volatile condensable substances which will be released by a polymeric material at 125°C in vacuo. (It will not illustrate the subsequent evaporation of condensed material with time, which is indicated by the VCM curves established for engineering information.) Although the concept will (hopefully) qualify as a specification procedure for screening purposes, it will also provide invaluable engineering information where thick and/or protected polymeric materials are involved. For example, a flange sealant material, which will have very little exposed surface area, may not display large VCM values in the 300-hr thermal-vacuum test periods; however, a large maximum-VCM value may indicate that a tremendously long time of evolution of material must be anticipated.

Results of studies of the effects of a vacuum-thermal environment on the mechanical properties of silicone rubbers SE-555 and -3604 and Hycars-1 and -3 are presented in Tables V and VI. In addition, Table VII contains the results of stress relaxation tests of the silicone rubbers in air at 125°C. As indicated in the Tables, Hycar-1 appears to perform better than Hycar-3. Although SE-555

TABLE V

EFFECT OF VACUUM-THERMAL ENVIRONMENT ON STRESS RELAXATION BEHAVIOR OF HYCAR AND SILICONE RUBBERS

Material	Intermittent			Continuous		
	Approx. Time to $f(t)/f(t)>1.0$, hours	$f(t)/f(o)$ at		Approx. Time to $f(t)/f(o)=0.8$, hours	$f(t)/f(o)$ at	
		20 hrs	500 hrs		20 hrs	500 hrs
SE-555 Red	2	1.06	1.36	1000	0.87	0.82
SE-3604	2	1.04	1.21	---	0.92	0.89
HYCAR-1	1	1.16	~1.65	450	0.84	0.80
HYCAR-3	3	1.06	1.38	2	0.59	0.48

Notes:

1. All tests conducted at strains of approximately 0.25
2. Data obtained from best curves drawn through duplicate test results
3. Exposure conditions consisted on two stages:

   SE-555 and -3604 -(a) 280 hours at $50^{\circ}C$ and an average pressure of about 4×10^{-6} torr (b) 1040 hours at $125^{\circ}C$ and average pressures of from 3.2 to 4×10^{-6} torr.

   HYCAR-1 and -3 -(a) 215 hours at $50^{\circ}C$ and an average pressure of about 4×10^{-6} torr.

TABLE VII

EFFECT OF VACUUM-THERMAL ENVIRONMENT ON TENSILE PROPERTIES OF HYCAR AND SILICONE RUBBERS

Material	History	Test Temp., °C	Stress at Strain of 0.25, psi	Stress at Rupture, psi	Stress at Rupture, in./in.
SE-555 Red	Control	25	65	825	6.30
	Exposed	25	84	760	4.35
	Control	125	53	310	2.94
	Exposed	125	77	311	1.95
SE-3604	Control	25	109	637	1.59
	Exposed	25	131	662	1.60
	Control	125	112	405	0.99
	Exposed	125	125	463	1.03
HYCAR-1	Control	25	95	1180	2.77
	Exposed	25	166	990	1.48
	Control	125	75	385	1.46
	Exposed	125	155	404	0.77
HYCAR-3	Control	25	104	1240	1.94
	Exposed	25	184	980	0.98
	Control	125	108	435	1.06
	Exposed	125	192	505	0.63

Notes:
1. All data points are averages of measurements on duplicate specimens.
2. Tests were conducted at an extension rate of 0.1 in./min.
3. Control specimens were stored at normal room conditions for the entire period from specimen preparation to final testing.
4. Exposure conditions were as indicated in Table V.

TABLE VII

EFFECT OF AIR-THERMAL ENVIRONMENT ON STRESS RELAXATION BEHAVIOR OF SILICONE RUBBERS

Material	Intermittent		Continuous	
	Approx. Time to $f(t)/f(o) > 1.0$, hours	$f(t)/f(o)$ at 90 hours	Approx. Time to $f(t)/f(o) > 0.8$,	$f(t)/f(o)$ at 20 hours
SE-555 Red	> 91	1.0	27	0.81
SE-3604	>118	1.0	>118	0.92

Notes:

1. All tests conducted at strains of approximately 0.25.
2. Data obtained from tests of single specimens.
3. Environment consisted of one atmosphere of air at 125°C.

appeared to be superior in most respects to all other elastomers tested thus far, SE-3604 showed even better performance. The tensile properties of rings of SE-3604 stored in the vacuum-thermal environment changed so little that one might report no change.

Work is in progress in preparation for studies of the influence of sterilization cycling and the vacuum-thermal environment on Lexan. These studies will involve tests of the material under constant load. Current tests involve studies of reproducibility of constant load failure and the establishment of maximum allowable loads.

Most of the equipment is now on hand for two prototype long-term mechanical-property measurement units. The base plates have been machined and provided with ports for ion-pump attachments and lead-through electrical connections. Work is underway on fabrication of constant strain and load fixtures for the polymeric samples and toward establishing of localized heating (by an inner cylinder with bands of electrically-conducting glass); samples are being prepared for the tests.

Work has proceeded on the examination of polymers in the infrared and far infrared regions of the electromagnetic spectrum. However, it has been found that the carbon-filled polymers, such as Hypalon and Viton, will not transmit sufficient light energy in the far infrared to operate detectors even though extremely thin specimens have been prepared with a microtome. Continued efforts will involve examination by attenuated total reflectance (ATR) techniques.

The modification of one of SRI's mass spectrometers to provide for higher resolution and sensitivity is now complete; subsequent to current calibration and check-out, work will continue on the identification of substances released by polymeric materials *in vacuo* at 125°C.

FUTURE WORK

Work will be accelerated toward the installation and operation of the assembly for rapid maximum VCM determinations. Fabrication of support structures for the VCM clam-shell units will continue.

Construction of long-term mechanical property units will be completed, and it is anticipated that heat-sterilization studies will commence shortly.

Work will continue on the spectral studies of polymers exposed to a thermal-vacuum environment and on the measurements of changes in mechanical properties of polymers in a thermal-vacuum environment.